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Physico-Chemical Properties of W Metal Powder

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19. Physico-Chemical Properties of W Metal Powder

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The structure of W metal powder obtained from the oxide by H_2 reduction is complex. In report I (N. Sasaki, R. Ueda: Science of Powder. 3. (1949) 1) a viewpoint was proposed for the clearer understanding of the structure, namely, the ultimate unit is W single crystals of various shapes ranging from about 0.5μ to 0.1μ . These "primary particles" partly exist as such, but are largely found as aggregate with some solidity. These "secondary particles" in their turn are partly found in loosely connected "tertiary particles" and so on.

The present paper is concerned with the effect of the pressing on the structure of powder produced from two different sorts of oxide, the one being oxide obtained by the decomposition of sodium tungstate with hydrochloric acid, the other by roasting ammonium-paratungstate. The reduction was carried out by continuously elevating the temperature from 500 to $800^\circ C$ in about one hour. The powder was pressed to a briquette under hydraulic pressure of 11 tons per square inch. In water the briquette disintegrated at once and on slight stirring dispersed to fine particles. With the dried powder and the original powder following measurements were made: a) the specific gravity calculated from the sedimentation volume; b) the surface area per gram by B.E.T. method and the mean particle diameter calculated therefrom; c) the particle size distribution by Wiegner; and d) electron microscopic observations.

The results are shown in accompanying table.

W powder from	pressing	sp. grav.	Surface area per gram	mean dia from B.E.T.	particles size distribution	
					Wiegner method μ	elect. mic- roscope μ
WO ₃	before	4.0	0.651m ² /g	0.476	1.5~3.5	0.2~2
	after	7.5	0.658 "	0.473	0.5~1.5	0.2~2
amm. para- tungstate	before	4.0	0.25 "	0.497	3~14	0.5~3
	after	9.1	0.631 "	0.492	0.5~3	0.3~3

From that results we see that

- 1) The pressing greatly reduced the particle size of the original powder (the

electron microscopic observation reveals this to lesser degree, probably due to inadequate dispersion of particles in the specimen) and halved the sedimentation volume, while the surface area or the mean particle diameter remained almost the same. This clearly shows that the pressing could almost completely disintegrate the secondary and tertiary particles to the primary particles but produce no fresh surface by deforming or crushing the primary particles themselves.

The metallurgical importance seems, therefore, to lie rather in the size distribution of the primary particles as revealed by the powder subjected to pressing than in the distribution of the original powder.

20. On the Precipitates of Tungstic Acid

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The precipitates of tungstic acid produced by adding sodium tungstate solution to hydrochloric acid were observed with an electron microscope and dehydration curves of these precipitates were obtained by the use of a quartz-fibre spring balance.

(1) A turbid solution obtained by adding 0.5 ml of 0.1 molar sodium tungstate solution to 10 ml of 0.05 molar hydrochloric acid at room temperature, contained on centrifugifying the precipitate (a) consist of thin crystals of various forms, round, semi-elliptical, square, boat-like and needle-like ($0.2\text{--}2\mu$). These crystals, if left in solution, slowly form aggregates which hardly disperse on addition of water. The supernatant solution contained fine needles (0.1μ) and small granules (0.2μ) which on standing assumed respectively the form of network and threads.

(2) The precipitate (b) produced by pouring sodium tungstate solution into hot hydrochloric acid consists of very fine angular plates (0.05μ).

(3) The dehydration curves are continuous with precipitate (a) and discontinuous with precipitate (b) whose composition is $\text{WO}_3 \cdot \text{H}_2\text{O}$ at $85\text{--}185^\circ\text{C}$.

(4) Strong electron beam or heating decomposes thin crystals of tungstic acid to small granules randomly scattered within their original forms.

21. Influence of Slag, especially of Al_2O_3 and TiO_2 in Slag upon the Structure and Mechanical Properties of Cast Iron. (V)

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The gray cast iron was melted under the slag of $\text{SiO}_2\text{--CaO--Al}_2\text{O}_3\text{--TiO}_2$ system